

Quantifying the Available Soil Phosphorus Pool with the Acid Ammonium Oxalate Method

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ABSTRACT

Measurement of the available soil P pool is required for making P fertilization recommendations. Extractants capable of estimating this P pool are needed. The objective of this study was to explore the utility of the acid ammonium oxalate method for estimating available soil P pools in diverse soils. Oxalate extraction in the dark dissolves mainly active noncrystalline Fe and Al in the soil. Oxalate extractable P (P_{ox}) has rarely been investigated for predicting the available P pool in highly weathered soils, where Fe and Al oxides are usually abundant. We estimated P_{ox} in five highly weathered soils and three slightly weathered soils amended with varying amounts of P. The size of the available P pool in each soil was also estimated by a modified Hedley sequential extraction and verified in a 14-crop exhaustive greenhouse experiment. Results indicated that P_{ox} in all soils appeared to be largely plant-available. P_{ox} was found unsuitable for quantifying the available P pool in the calcareous, slightly weathered soils. Carbonates raised the pH of the oxalate solution and reduced the concentration of the oxalate anion to differing degrees, thus making this method unreliable for quantifying available P pools in calcareous soils. In highly weathered soils, the relationship between the quantity of available P and P_{ox} was described by a linear equation, $Y = 294.7 + 1.05 X$ ($r^2 = 0.81$), where Y is estimated available P in mg P kg⁻¹, and X is P_{ox} in mg P kg⁻¹. The P_{ox} was a good predictor of the available P pool in the highly weathered soils that were measured by the Hedley procedure and a multicrop exhaustion experiment.

ACTIVE NONCRYSTALLINE Fe AND Al CONSTITUENTS are important in determining P availability in many soils because of their large surface areas (Hodges and Zelazny, 1980) and high reactivity with phosphates (Saunders, 1964). Phosphorus sorption capacity of most highly weathered soils can be attributed to P sorption capacity of various oxides, hydroxides, and oxyhydroxides of Fe and Al (van der Zee and van Riemsdijk, 1986; Parfitt, 1989; Borggaard et al., 1990), and some of these components are active noncrystalline Fe and Al. Active noncrystalline Fe and Al oxides in the clay fraction and in whole soils have been estimated by acid ammonium oxalate (pH = 3) extraction in the dark (Hodges and Zelazny, 1980; Borggaard, 1992; Lookman et al., 1995). Analogous to the extraction of active noncrystalline Fe and Al in soils, phosphorus associated with noncrystalline Fe and Al has also been extracted by oxalate (van der Zee et al., 1987; Wang et al., 1991; Freese et al., 1995; Lookman et al., 1995). Yuan and Lavkulich (1994) found that oxalate extractable P represented 17 to 66% of the total P sorption capacity in 43 Spodosols. In highly weathered soils, such as Oxisols and Ultisols in tropical

environments, the contribution of active noncrystalline Fe and Al to P sorption capacity may be even greater than in Spodosols. Estimation of the quantity and plant availability of P associated with active noncrystalline Fe and Al is, thus, significant.

Previous extractants were developed for two major reasons (Kamprath and Watson, 1980): (i) to estimate a fraction of soil P and to correlate it with plant availability; and (ii) to separate deficient from nondeficient soils. These extractants fail to estimate directly the amount of P available to plants. Studies have suggested that extractable P is buffered by some pools (Novais and Kamprath, 1978; McCollum, 1991). The buffering pools, as well as the total quantity of the general available pool, however, have seldom been quantified.

Oxalate extractable P includes noncrystalline Ca-, Al-, and Fe-P (Wang et al., 1991). These noncrystalline P components were found to be more easily extractable by Olsen (0.5 M NaHCO₃, pH 8.5) and Mehlich 1 (0.05 M HCl + 0.0125 M H₂SO₄) extractants than was P that was associated with crystallized minerals (Wang et al., 1991). These findings imply that P associated with noncrystalline materials may be more plant available than P associated with crystalline P minerals. The P_{ox} in a group of sandy European soils was considered reversible during a long-term desorption study (Lookman et al., 1995). Freese et al. (1995) also suggested that the P_{ox} in six Belgian sandy soils was highly reversible, as indicated by the absence of a desorption plateau after >500 h of continuous P removal by a hydrous ferric oxide sink. In highly weathered soils where Fe and Al oxides are abundant, the reversibility or plant availability of P_{ox} is not easily predicted.

In a recent study seeking to predict residual available P in highly weathered soils, P extracted by iron-impregnated strips (Strip-P), NaHCO₃-, and NaOH-, and HCl-P extracted from a modified Hedley method appeared to buffer available P under exhaustive plant removal of soil P. These fractions were grouped into a potentially available P pool (Guo and Yost, 1998). Because oxalate is more selective for dissolving primarily noncrystalline Fe and Al oxides than is dilute NaOH (Hodges and Zelazny, 1980), P_{ox} may be a more accurate representation of P associated with noncrystalline Fe and Al in highly weathered soils than is NaOH-P in the Hedley method. Oxalate also partially dissolves P that is extracted by the iron-impregnated paper strips, NaHCO₃, and dilute HCl. This may be owing to the following explanations. The oxalate anion replaces P sorbed at metal-hydroxide surfaces through ligand-exchange reactions, and it complexes metals in solution and thus prevents precipitation of metal phosphates

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Table 1. Soil taxonomy and selected chemical properties of the eight soils studied.

Soil series	Subgroup	pH (1:1 H ₂ O)	Carbonates (as CaCO ₃)†	Fe _{ox} ‡	Al _{ox} ‡	Total P§	Amorphous content¶	Oxalate extractable P‡
				g kg ⁻¹				
						mg kg ⁻¹		
Honouliuli	Typic Chromustert	7.26	3.2	3.40	1.43	1.840	24.0	883.8
Lualualei	Typic Chromustert	7.65	16.2	3.82	2.94	2.098	13.4	1685.6
Nohili	Cumulic Haplaquoll	7.44	65.8	11.80	4.32	0.815	25.9	398.9
Paaloa	Rhodic Kandudult	5.05	--#	7.48	2.98	0.596	63.9	77.8
Wahiawa	Rhodic Eutruxox	5.05	--	3.27	3.65	0.528	41.3	152.9
Kapaa	Anionic Acrudox	4.92	--	4.54	5.61	1.722	72.2	93.5
Leilehua	Ustic Kanhaplohumult	4.66	--	6.56	6.50	0.699	79.6	131.9
Mahana	Humic Rhodic Haplustox	4.31	--	5.70	12.81	1.327	91.7	579.2

† Determined by the method of Nelson (1982).

‡ Acid ammonium oxalate extraction (pH 3) (Hodges and Zelazny, 1980).

§ Na₂CO₃ fusion (Olsen and Sommers, 1982).

¶ By the method of Hodges and Zelazny (1980).

Negligible.

(Fox et al., 1990). Readsorption of dissolved P in the oxalate extract is, therefore, less likely than in most conventional extractants, because of the formation of complexed Fe and Al, and because Ca precipitated by the oxalate anion causes less precipitation and readsorption of phosphate as Ca-P (Fox et al., 1990; Borggaard, 1992). The acidity of oxalate makes its mode of extraction similar to that of HCl. These characteristics of oxalate extraction led us to hypothesize that the available soil P pool, as measured by the multistep Hedley sequential extraction procedure, may be estimated by P_{ox}. Estimating the size of the available P pool in soils would be very useful for calculating P-use efficiencies as related to agronomic practices and for assessing soil P that represents an environmental hazard. Based on the findings above, the objective of this study was to determine if oxalate can be used as an extractant for estimating the available P pool in soils.

MATERIALS AND METHODS

Soils

Samples of eight soils were collected from the surface 0- to 15-cm layer in Hawaii. The major criterion for selecting these soils was to represent a diversity in weathering and a wide range in P sorption capacity. Background soil information is described in Table 1. Phosphorus sorption capacity of the soils (Table 2) was determined by the Fox and Kamprath (1970) method. Soils were grouped according to their P sorption isotherms (not shown). The P application rates (Table 2) were chosen so that no additional P would be needed during the exhaustion experiment, and so changes of P fractions due to depletion could be observed.

Table 2. Phosphorus application rates for the greenhouse experiment.

Soil series	P applied to achieve target solution P levels (mg L ⁻¹ P)†			
	Control	0.2	0.5	1.0
mg kg ⁻¹ P				
Low P sorption Honouliuli, Lualualei, and Nohili	0	100	250	500
Medium P sorption Paaloa and Wahiawa	0	500	650	800
High P sorption Kapaa, Leilehua, and Mahana	0	1400	1800	2200

† Determined by the Fox and Kamprath (1970) method.

Greenhouse Experiment

Soils were air-dried and ground to pass a 4-mm sieve using a mechanical grinder. Acidic soils were limed to pH 6.5 by adding laboratory grade, powdered CaCO₃ according to the Adams and Evans (1962) buffer method. After liming, the soils were moistened to their available water capacity (measured as soil water between matrix potential of -0.03 MPa and -1.5 MPa) and incubated for 4 wk in plastic bags. Five-kilogram (dry wt. basis) portions of each soil were weighed onto clean brown paper. The prescribed amount of P (Table 2) as Ca(H₂PO₄)₂·H₂O was thoroughly mixed with soil. Treated soils were then placed into plastic pots (18 cm in diameter, 30 cm in depth). Soils were then watered weekly with deionized water to available water capacity and allowed to dry in the greenhouse through four weekly cycles. Sixty days after P application, 50 mg kg⁻¹ N as urea, 60 mg kg⁻¹ K as KCl, 25 mg kg⁻¹ Mg as MgSO₄·7H₂O, and 5 mg kg⁻¹ Zn as ZnSO₄·7H₂O were added to each pot and mixed.

The experimental design was a randomized complete block with three replicates of each soil. Corn (*Zea mays* L.) was grown for Crops 1 to 10, 13, and 14. Soybean [*Glycine max* (L.) Merr.] was grown for Crops 11 and 12 because corn was not growing as well as it had previously. When corn was grown, each pot received 12 pregerminated corn seeds (Hybrid X304CF15, Pioneer Hi-Bred Int., Johnston, IA), which were placed about 1 to 2 cm below the soil surface. At 3 d after emergence (DAE), each pot was thinned to 10 seedlings. Two additional doses of N at 50 mg kg⁻¹ were applied at 10 and 20 DAE. Plants were grown for 4 wk.

For the two soybean crops, five healthy, noninoculated, and presoaked seeds (cv. Kahala, Dep. of Horticulture, Univ. of Hawaii) were placed 1 to 2 cm below the soil surface in each pot and thinned to three seedlings at 3 DAE. Soybean plants were grown for 45 d. Nitrogen was applied at 56 mg kg⁻¹, and equally split between 0 and 15 DAE. Boron (as sodium borate) and molybdenum (as ammonium molybdate) were applied at 0.9 mg kg⁻¹ to the first soybean crop. The pots were watered daily with deionized H₂O and moisture was maintained near soil available water capacity throughout the growth period.

At harvest of each crop, plants were cut at the soil surface, washed with deionized H₂O, and dried at 70°C. Each pot was emptied and the soil was crushed on a piece of brown paper. A 2-g subsample was taken. Roots were removed with the soils in the same manner as described by Fixen and Ludwick (1982).

Dried plant tissue was ground to pass a 0.5-mm sieve. Tissue P was determined using a dry oxidation procedure. Subsamples weighing 0.05 to 0.10 g were ashed at 500°C for 4 h. The ash was dissolved in 1 mL 1 M HCl and diluted to 10 mL with deionized water. Inorganic P was determined by the method of Murphy and Riley (1962).

Soil Phosphorus Fractionation

Soil samples collected after Crop 0, 2, 4, 6, 8, 10, 12, and 14 were air-dried, and ground to pass a 0.25-mm sieve. The samples were then sequentially extracted for P using the Hedley procedure (Hedley et al., 1982) modified in this laboratory as follows. Triplicate 0.5-g samples of soil were weighed into 40-mL screw-capped centrifuge tubes, and the appropriate extractant was added and the tubes shaken for 45 min of each hour for 16 h. The extractants, in sequential order, were: (i) 30 mL deionized water and one 2 cm by 10 cm Fe-impregnated filter paper strip as prepared according to Guo et al. (1996); (ii) 30 mL of 0.5 M NaHCO₃ at pH 8.5; (iii) 30 mL of 0.1 M NaOH; (iv) 30 mL of 1 M HCl; and (v) 5.0 mL of conc. H₂SO₄ (18 M) with 2 to 3 mL H₂O₂ (30%) in 0.5-mL increments. Inorganic P (P_i) in the extracts was determined by the method of Murphy and Riley (1962) after pH adjustment using β -nitrophenol as an indicator. Total P (P_T) was measured in both the NaHCO₃ and the NaOH extracts for calculating organic P (P_o). Total P was determined with the Murphy and Riley (1962) method after autoclave digestion to convert P_o to P_i with acid ammonium peroxydisulfate (APHA, AWWA, and WEF, 1995). P_o was calculated as the difference between P_T and P_i in the extracts.

Oxalate Extractions of Soil Phosphorus

Subsamples of soils prepared for fractionation were also extracted with acid ammonium oxalate. Acid ammonium oxalate (0.2 M) was prepared by dissolving 65.53 g oxalic acid and 96.65 g ammonium oxalate in 6 L deionized water (U. Schwertmann, 1996, personal communication). Solution pH was adjusted to 3.0 with dilute HCl or NH₄OH. Phosphorus was extracted by adding 30 mL of 0.2 M ammonium oxalate solution to duplicate 0.5-g soil samples in screw-cap centrifuge tubes. The sealed centrifuge tubes were placed immediately inside a covered box to eliminate light and shaken for 2 h. After shaking, the tubes were centrifuged at 11 950 \times g rpm for 10 min, and the supernatant from each tube was decanted into a plastic tube. An aliquot (usually 1 mL) of the extract was evaporated to dryness in a heated oven. The residue was ashed at 500°C for 1 h (Wang et al., 1991), and redissolved in 1 M HCl. Phosphorus was determined by the method of Murphy and Riley (1962). Data were processed using SAS for Windows, version 6.11 (SAS, 1996).

RESULTS AND DISCUSSION

Highly Weathered Soils

Availability of Oxalate Extractable Phosphorus

In all soils, P_{ox} declined with plant P removal (Fig. 1). Because the patterns of change were similar for all P application rates, only results from soils treated with P to raise soil solution P to 0.2 mg L⁻¹ are shown. The Hedley residual P in the five highly weathered soils was observed to accumulate gradually during exhaustive plant P removal (Guo and Yost, 1998). The decline in P_{ox} may be caused by either plant P removal or transformation of P_{ox} to less available P forms. The current study did not resolve the importance of these two processes relative to the overall decline of P_{ox}. The relationship between the decline in P_{ox} measured after every two consecutive crops and the cumulative plant P uptake of the corresponding two crops was analyzed. Oxalate extractable P in soils receiving three P rates (except for

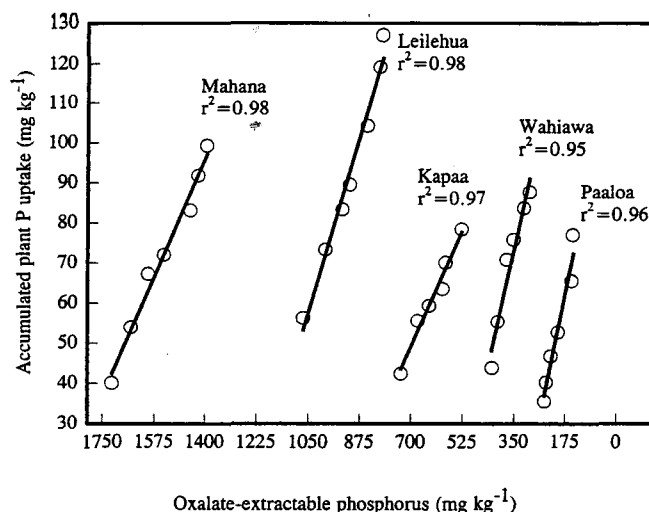


Fig. 1. Relationships between accumulated plant P and oxalate extractable P in five highly weathered soils. Phosphorus was added to raise soil P to 0.2 mg L⁻¹ in 0.01 M CaCl₂.

the control) in all 14 crops was included in the analysis. The high correlation between P_{ox} decline and cumulative P uptake indicated that P_{ox} was related to plant P removal (Table 3). A high plant P uptake was associated with a large decline in P_{ox} and low uptake was associated with a small decline in P_{ox}. The probability that the decline was due to plant P removal was high (Table 3). The decline in P_{ox} was not significantly correlated with the increase in residual P (data not shown). Thus, the high correlations between plant P uptake and P_{ox} suggest that P_{ox} estimates plant available P. Results of this study were consistent with the findings by Lookman et al. (1995), which suggested that all P_{ox} was potentially desorbable in sandy soils. Wang et al. (1991) also provided evidence that a higher proportion of P_{ox} was dissolved by Olsen (0.5 M NaHCO₃, pH 8.5) and Mehlich 1 (0.05 M HCl + 0.0125 M H₂SO₄) extractants than was P that is associated with crystallized minerals.

Oxalate Extractable Phosphorus vs. Available Phosphorus Estimated by the Hedley Method

Available P in the five highly weathered soils was estimated by including all P fractions except residual

Table 3. Correlation coefficients between the decline in oxalate-extractable P measured after every two consecutive crops and P uptake during the corresponding two crops.

Soil series	Coefficients	Probability
Highly weathered soils†		
Paaloa	0.942	0.0001
Wahiawa	0.810	0.0001
Kapaa	0.778	0.0001
Leilehua	0.889	0.0001
Mahana	0.657	0.0031
Slightly weathered soils		
Honouliuli	0.885	0.0001
Lualualei	0.315	0.1025
Nohili	0.279	0.2343

† Data from the control (no added P) of Paaloa, Wahiawa, Kapaa, Leilehua, Mahana, and Nohili were not included in calculating coefficients because of both low native P and low plant P uptake.

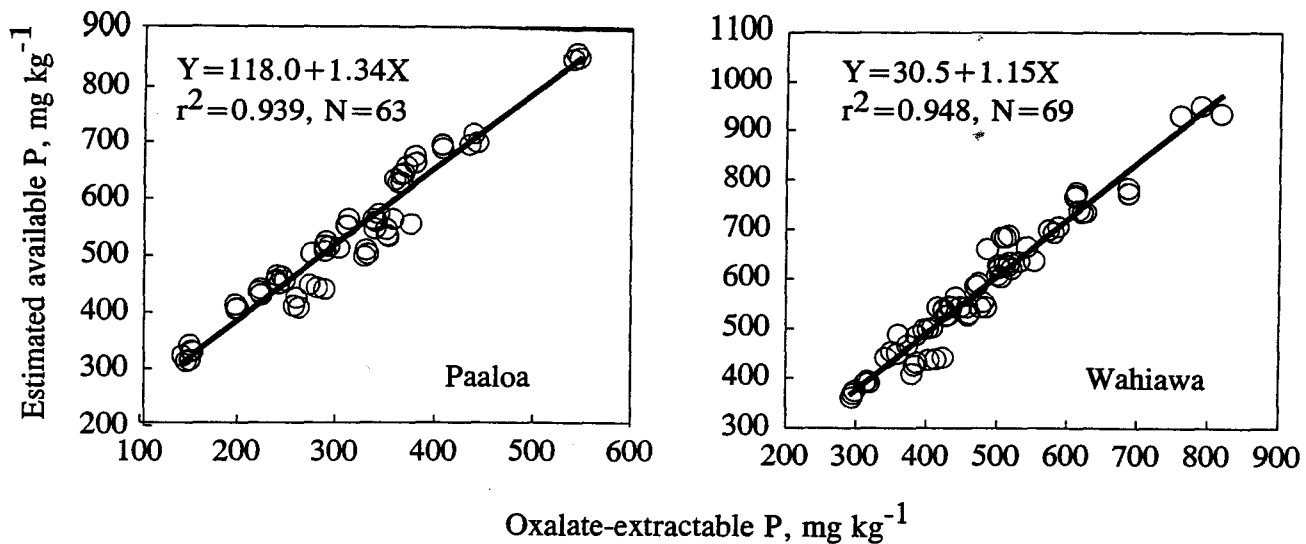


Fig. 2. Relationships between oxalate extractable P and available soil P estimated from a modified Hedley procedure in two highly weathered soils of medium P-sorption capacity. Available data from all crops at all P rates were plotted.

P in the modified Hedley procedure. The estimated available P was linearly related to P_{ox} for each soil (Fig. 2 and 3). In the Paaloa, Wahiawa, and the Leilehua soils, P_{ox} accounted for >90% of the variation in estimated total available P by the Hedley method; in the Kapaa and the Mahana soils, >80% of the variation in estimated available P was also explained by P_{ox} (Fig. 2 and 3). The estimated total available P by the Hedley method in all five highly weathered soils could be predicted by P_{ox} with a linear equation, $Y = 294.7 + 1.05X$, where Y was estimated available P from the Hedley method in mg kg⁻¹, X was P_{ox} in mg kg⁻¹ (Fig. 4). More than 80% of the variation in these soils with three P rates during the entire sequence of 14 consecutive crops was explained by this equation.

The Hedley procedure requires seven separate measurements for estimating the P fractions. Interpretation of results involving all fractions are often complex. Re-

sults of this study suggested that the available P pool in the highly weathered soils may be estimated by P_{ox} (Fig. 4). Among the potentially available fractions of the Hedley procedure, the Strip P and $NaHCO_3$ -P_i may be P that is primarily bound to the reactive surfaces that are in direct contact with the aqueous phase, as suggested by Hingston et al. (1974), or as "more physically adsorbed phosphate" (Sharpley, 1991). The $NaHCO_3$ - and $NaOH$ -P_i may be P that is associated with organic materials or organic matter and Fe and Al complexes (Schoenau et al., 1989). The $NaOH$ -P_i probably represents P that is associated with noncrystalline Fe and Al oxides (Hedley et al., 1982; Tiessen et al., 1984; Wager et al., 1986).

In addition to the ability to remove P sorbed by noncrystalline Fe and Al (van der Zee et al., 1987; Wang et al., 1991; Lookman et al., 1995) oxalate as an anion also replaces P on oxide surfaces through ligand ex-

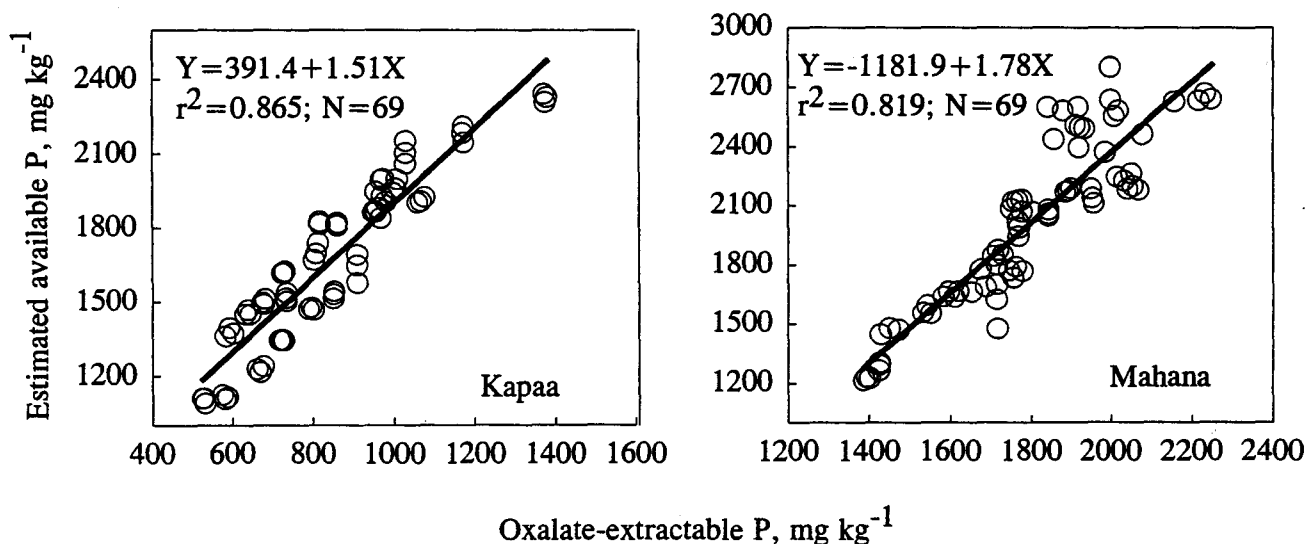


Fig. 3. Relationships between oxalate extractable P and estimated available soil P from a modified Hedley procedure in two highly weathered soils of high P sorption capacity. Available data from all crops at all P rates were plotted.

change (Fox et al., 1990) and extracts most organic Fe (Schwertmann, 1973) and organically bound Al (Yuan and Lavkulich, 1994). Thus, it is not surprising that P_{ox} in the five highly weathered soils was similar to the available P estimated by the Hedley procedure as indicated by the 1:1 line (Fig. 4). Although P_{ox} closely estimated available P in the highly weathered soils, it is possible that P_{ox} may overestimate P that is otherwise unavailable to plants, such as P covered by noncrystalline Fe and Al. On the other hand, oxalate may also leave behind undissolved P bound with noncrystalline oxides that are otherwise plant available, or P bound with noncrystalline Fe and Al and protected or hidden in nodules and concretions (Borggaard, 1992).

Slightly Weathered Soils

In contrast with the highly weathered soils, P_{ox} in the three slightly weathered soils did not appear to be a good predictor of available P as estimated by the modified Hedley procedure. The decline in P_{ox} measured after every two consecutive crops and the cumulative plant P uptake of the corresponding two crops was not significantly correlated in the Lualualei and the Nohili soils (Table 3), which are both calcareous (Table 1). The poor correlation suggested that the decline in P_{ox} may not necessarily be due to plant P removal in two of the three slightly weathered soils. In addition, a single straight line did not describe the relationship between P_{ox} and the available P estimated by the modified Hedley procedure. In the Honouliuli and the Lualualei soils, only 55 to 58% of the variation in the estimated available P could be explained by P_{ox} (Fig. 5). Although more of the variation in estimated available P in the Nohili soil was accounted for by P_{ox} , the correlation between the decline in P_{ox} measured after every two consecutive crops vs. cumulative P uptake during the corresponding two crops was poor (Table 3). Thus, P_{ox} did not adequately predict the quantity of available P estimated by

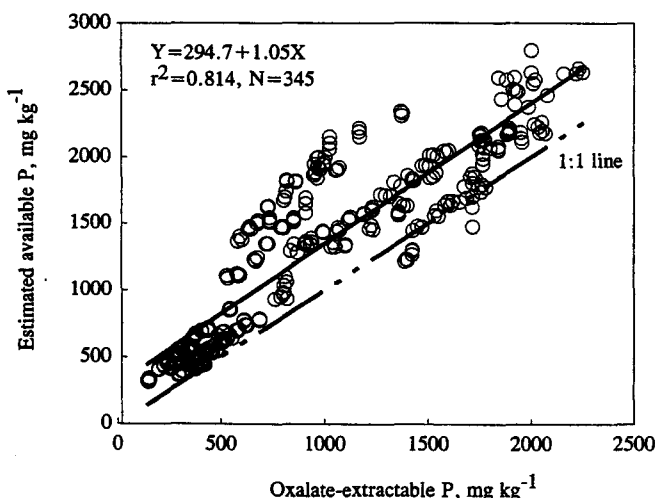


Fig. 4. Relationships between oxalate extractable P and estimated available soil P from a modified Hedley procedure in five highly weathered soils. Available data from all crops at all P rates were plotted.

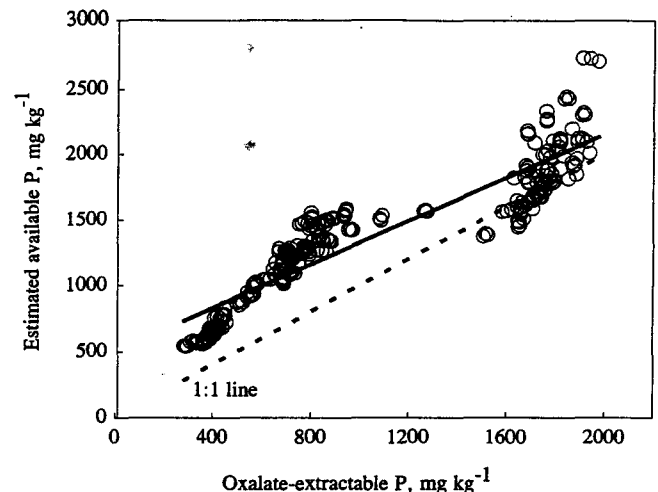


Fig. 5. Relationships between oxalate extractable P and estimated available soil P from a modified Hedley procedure in three slightly weathered soils. Available data from all crops at all P rates were plotted.

the modified Hedley method in the slightly weathered soils.

The inadequacy of the oxalate method on the slightly weathered soils was probably due to three factors: (i) high soil alkalinity that raises the pH of the extractant; (ii) high soil Ca that reduces the oxalate anion; and (iii) extraction of Ca-P that is not readily available to plants. The three slightly weathered soils contained free carbonates (Table 1). X-ray diffraction results revealed that the Lualualei soil had the highest percentage of calcite of the three soils; the Nohili had both calcite and dolomite (data not shown). The pH of the oxalate extractant was found to rise above 4 after a 2-h extraction in these soils (Parfitt, 1989). This pH increase may lead to a decrease in the amount of oxides extracted compared to that at pH 3 (Parfitt, 1989). In addition, the dissolved Ca^{2+} in the extract may precipitate oxalate during extraction, which reduces oxalate concentrations to $<0.2 M$ in the original extract. These complications may explain the poor correlation between the decline in P_{ox} and P uptake in the Lualualei and Nohili soils, which contained calcite. When measuring active noncrystalline Fe and Al in some calcareous soils, decalcifying procedures have been used (Loeppert and Hallmark, 1985; Morris et al., 1990; Cordero del Campillo and Torrent, 1992). However, these procedures further complicate the P_{ox} extractions because the decalcifying reagent will also attack soil P. Use of P_{ox} in estimating available P in calcareous soils remains unreliable.

CONCLUSIONS

Our results indicated that oxalate extractable P in the highly weathered soils declined when soil P was removed by continuous cropping. The decline in P_{ox} measured after every two consecutive crops in a greenhouse trial and the cumulative plant P uptake during the corresponding two crops was highly correlated, indicating that P_{ox} responded to the withdrawal of available P. Available P estimated with a modified Hedley proce-

ture can be approximated by the P_{ox} in each of the five highly weathered soils by a linear equation with an r^2 of 0.81. In contrast, P_{ox} in the slightly weathered soils was inadequate for estimating available P. The decline in P_{ox} measured after every two consecutive crops and the cumulative P uptake during the corresponding two crops generally was not significantly correlated with plant P uptake in the calcareous soils. The carbonates in calcareous soils reduce acidity and oxalate concentration during extraction. Oxalate also dissolves some Ca-P that is usually not readily available to plants. These complications in methodology may explain the poor correlation between the decline in the P_{ox} and P uptake in the calcareous Lualualei and Nohili soils. The oxalate method remains unusable for estimating available P pools in calcareous soils. The successful use of acid oxalate for estimating available soil P in highly weathered soils, however, shows promise, and this extractant may be considered for estimating available soil P pools in noncalcareous soils.

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